Construction of a High-Pressure Viscometer-Operating Manual

Final Report to

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Introduction

The limiting low-shear viscosity of liquid lubricants at high pressures is of interest to Timken. The pressure-viscosity behavior of liquids is essential for the calculation of film thickness and friction in lubricated concentrated contacts. Fine details of chemical structure can produce significant differences in viscosity at high pressure [1].

Principle of Operation

Nearly all viscometer configurations have been pressurized. For simplicity and accuracy in measuring low-shear viscosity, however, the falling body type is preferred. This configuration was perfected by Bridgman. The falling body is a cylindrical sinker with guiding lugs provided in cruciform arrangement at each end. The sinker falls within a close fitting cylindrical tube with minimal pressure difference across its thickness. Bridgman detected the beginning and end of a fall by mechanically switching an electric circuit with the sinker. Others have inductively detected the end points. In this laboratory the position of the sinker is continuously monitored using a linear variable differential transformer, LVDT, by constructing the sinker from magnetic material and the pressure vessel from non-magnetic material. The cylindrical bore surrounding the sinker is a non-magnetic tube, plugged at one end and fitted with a volume-compensating piston at the other. The tube, sinker, plug and piston constitute the viscometer cartridge which is removed from the vessel for exchanging sample and sinkers. A sinker may be provided with a through hole to increase the falling velocity and the relationships between geometry, viscosity, density and falling velocity have been worked out; however in all applications the relationship between falling velocity and viscosity is found by calibration.

Specifications

A falling body viscometer has been constructed for Timken. This viscometer is based upon an instrument already in use in the Georgia Tech Center for High Pressure Rheology that was constructed in 2000 and has demonstrated good repeatability and reliability in ten years of use to 0.4 GPa pressure and to 170˚C temperature. At Georgia Tech, this instrument is used to characterize the viscosity at hydrodynamic pressures and at the pressure in the inlet sweep of the EHL contact. A new version of this viscometer was completed in 2003. The new design differs from others of similar capability in that the pressure vessel is not disassembled to access the

vessel chamber. Instead, a threaded plug provides easy access to the viscometer cartridge that resides within the pressure vessel.

The viscometer that was constructed as part of this program incorporates some design changes as a result of experience gained from use of the original version. A valve has been added to return the system to atmospheric pressure following a measurement series at elevated pressure and to introduce additional pressurizing medium at the start of a test series. A venting valve has been designed for this purpose instead and has operated properly throughout testing and calibration.

Operating specifications for the instrument are as follows:

A. Pressure to 400 MPa at 150˚C. A commercial pressure transducer with digital readout provides measurement with resolution of 1 MPa. Pressure is generated in the pressurizing medium (ethylhexyl sebacate) with an intensifier and hand pump. The calibration provided here gives an estimated accuracy of ± 0.5%. See Table 3. The entire system, less the pressure transducer, was pressurized to 500 MPa two times to condition the viscometer vessel. Operation at more than 425 MPa will overstrain the transducer.

B. Temperature to 150˚C. This upper temperature limit may be conservative. We have operated similar viscometers to 180˚C. Temperature reduces the life of the LVDT and promotes extrusion of the viscometer plug seal packing. The LVDT is rated for 150˚C by the manufacturer and we have observed a life of only several minutes at 220˚C. Viscometer temperature is measured with a type K thermocouple. The accuracy of measurement of sample temperature is believed to be ± 0.3˚C. Temperature of the viscometer is maintained and controlled by passing heated air through passages in the vessel. Operation requires air supply at 50 psig at 3.2 scfm.

C. Viscosity from 2 x 10^{-4} to 10^{4} Pa·s. The accuracy of the viscosity measurement for normal viscosities is believed to be ± 3 % (± 5 % for the hollow sinker), although repeatability of fall times may be as good as 1%. Three sinkers are provided to cover this range of viscosity. The lower viscosity limit for a sinker is reached when the fall distance required to reach terminal velocity becomes more than the space available for fall. For fall times greater than about 100s, accuracy is reduced due to instrumentation drift. The viscometer is provided with a signal conditioner for the

LVDT. Suitable recording instrumentation must be supplied by the user. A digital storage scope such as the Nicolet 420 is preferred and is common in most laboratories. The main requirement is for a record length adjustable from 2s to at least 4000s and a cursor option with time and voltage display. See more detailed requirements below.

D. Sample volume is less than 2 ml. Pressurizing medium, contained in the (green) charging pump, is di(ethylhexyl) sebacate or similar diester or PAO-4 with similar pressure-viscosity behavior to 0.4 GPa. The intensifier displaces 3.2 cm^3 at the high pressure end and 51 cm^3 at the low pressure end. The (yellow) pressurizing pump hydraulic oil was supplied by the pump manufacturer.

Oscilloscope Requirements

1) The LVDT signal must be represented in real time. The post-processing that is normal for computer-based scopes gives no indication of whether or not the operator has successfully begun a sinker drop. If the drop requires an hour, then an hour would be lost.

2) The scope must have a cursor for reading the time required for a certain change in LVDT signal.

3) The scope must have a sufficiently long time-base so that an event of at least 3000 seconds can be recorded. This will determine the largest valve of viscosity that can be measured. The 2090 model will record up to about 400,000 seconds.

Oscilloscopes found to be suitable

Nicolet model 420

Nicolet model 2090 with the 206 amplifier module (not the 204)

Nicolet model 4094

Norland Prowler

Viscometer Description

Overview

A section view of the viscometer proper is shown in Figure 1. A photograph of the viscometer and associated components, less the oscilloscope, is shown in Figure 2, including the yellow and green hand pumps and the control console. The components shown reside on a tabletop that measures 64 cm by 96 cm. It can be seen that this is absolutely the least space required and that a somewhat larger space is more convenient for arranging the various components. In the upper-right-corner of Figure 2, the air heating assembly must overhang the tabletop. This may be seen more clearly in Figure 3.

Vessel Plug

The distinctive difference between this viscometer and other LVDT based instruments is in the way that the viscometer is removed from the pressure vessel. See Figure 4 where the components which reside in the vessel are removed and shown in order of assembly. The vessel is closed at the top by the vessel plug that is part of an assembly containing a threaded nut, a thrust bearing and a seal shown at the top of Figure 4. The plug is removed from the vessel by unscrewing the nut which draws the plug and its seal out of the vessel. A viscometer cartridge is attached to the plug by a threaded fitting which is urged away from the plug by a spring to hold the cartridge at the bottom of the vessel.

Pressure Vessel

The pressure vessel is constructed of two concentric cylinders with heating air passages (grooves) in the interface between. They are permanently assembled by a vessel cover that contains external threads to engage the nut of the viscometer plug. A steel-jacketed thermocouple resides in the inner vessel near the sinker rest position. The two cylinders are assembled with 0.45 mm axial crush.

Cartridge Assembly

The cartridge is fitted with a removable threaded end plug at one end and internal threads to engage a spring loaded fitting on the vessel plug at the other. The viscometer cartridge, Figure

4, provides a bore into which the sinker fits. Pressure acts equally on the inside and outside of the cartridge tube so that the effect of pressure on the bore may be neglected. Changes in sample volume are accommodated by a compensating piston moving axially in the cartridge. A spring positions the cartridge at one end of the vessel. These parts are shown in the photograph of Figure 4 in order of assembly. All three sinkers, the cartridge assembly and cartridge related tools are shown in the provided wood box in Figure 5.

Sinkers

Three viscometer sinkers made of 430 stainless steel are provided: a solid, "S", sinker and a hollow, "H", sinker and a cup sinker, "C". Each has four guide lugs in cruciform arrangement at each end for centering in the cartridge tube. Formulae are available to calculate the relationship between falling velocity and viscosity; however, in use, this relationship is always found empirically. We find that the guide lugs, which are neglected in analytical solutions, contribute a significant portion of the viscous drag.

Viscosity, μ, is calculated from the fall time, t, the time required for a change of LVDT conditioned output of 100 mV.

$$\mu = Ct\frac{\rho_s - \rho}{\rho_s} \tag{1}$$

The estimated density of the liquid sample is ρ and the density of the sinker is ρ_s. An error of 10% in estimating ρ leads to only a 1% error in μ. The calibration factor, C, is found by experiment. Bridgman found that the change in calibration with pressure due to dimensional changes is less than 0.5% in 1 GPa and so the calibration is assumed here to be pressure independent. Properties of the sinkers and the applied shear stress, τ, can be found in Table 1. Spreadsheet programs are provided for data reduction for each sinker.

Sinkers have preferred axial orientations. The solid and cup sinkers have a bumper extending from the "bottom" and the hollow sinker has "T" ground into the side at the "top" end. The "bottom" refers to the end of the cartridge opposite to the spring where the sinker stops at the end of a fall. Bumpers reduce the squeeze effect at the end of a fall. The "H" sinker, lacking a bumper suffers from the central hole being obstructed by the end plug so that fall times must be measured far from the end of the fall, >= 150 mV in terms of LVDT signal.

Heating System

Heating air is supplied through a pressure regulator set to 23 psig and is mainly heated by a 600W (at 115 V) cartridge heater inside of a large tube upstream of the regulator shown in Figure 3. A custom-made rotary union (top of Figure 3) allows for rotation of the viscometer. A pressure switch in the control console disables the heater should the air supply to the regulator fall below about 40 psig. Temperature is adjusted by varying the supply voltage to the main heater. The required voltage, E, can be estimated from

$$E = K(T - T_{amb})^{\frac{1}{2}} \tag{2}$$

where T_{amb} is room temperature and K is approximately 9.2 volts/$^{\circ}C^{\frac{1}{2}}$. Final, precise adjustment of voltage is done manually by trial and error.

This system benefits from a new more precise closed-loop control of the temperature of the air supplied to the vessel. A 60W (at 115 V) cartridge heater is installed in the air supply upstream of the main heater and this heater receives the same manually regulated voltage as the main heater. The smaller auxiliary heater is controlled by a programmable controller which responds to the air temperature between the main heater and the vessel at the rotary union. A starting value for the air temperature setting can be found from

$$T_{set} = T + 0.17(T - T_{amb}) \tag{3}$$

Final, precise adjustment of air temperature setting is done manually by trial and error.

It is helpful to have a table of temperature settings that have been useful in previous measurements. Settings used in Atlanta are listed in Table 2.

Intensifier

The viscometer vessel is connected to an intensifier by a thick-wall tube. The intensifier has a theoretical pressure ratio (area ratio) of 16:1. Low pressure liquid is supplied through a rotatable quick-connect fitting (center right in Figure 2) to the large diameter piston end of the intensifier by a pressurizing hand-pump, the yellow pump. The high pressure medium (2-ethylhexyl sebacate) is replenished and the intensifier piston is reset by a green charging hand-pump (lower right in Figure 2) connected by a quick-connect fitting that connects to the venting valve for this purpose. A commercial strain-gauge pressure transducer, manufactured by GP-50, is attached by a threaded fitting and a ferrule to the side of the high-pressure cylinder. The low-

pressure end of the intensifier piston is sealed by an o-ring and the high-pressure end by an o-ring with a polymer back-up ring. A Bourdon tube gauge is provided at the (yellow) pressurizing pump. The pressure measured at this gauge, p_L, is related to the operating pressure, for increasing pressure (greater than zero) only by

$$p(\text{MPa}) = 0.103\ p_L(\text{psi}) - 5 \qquad (3)$$

with an accuracy of about 3 MPa. This relationship may be used to check the health of the pressure transducer.

The digital process indicator was scaled to read pressure in units of MPa using the factory calibration of the pressure transducer. A pressure calibration check is listed in Table 3 which indicates that the present pressure measurement is 3 MPa greater at 400 MPa than indicated from a 100,000 psi Bourdon-tube gauge from Heise. This gauge was employed because it is part of a large intensifier system capable of supplying a large volume of compressed medium. However, a 200,000 psi Hardwood manganin cell (Model C7186) when connected directly to the system with the Heise gauge also indicated 3 MPa higher than the Heise gauge at 400 MPa. The viscometer pressure measurement is therefore consistent with the newer Hardwood transducer. The viscometer is not capable of compressing a sufficient volume of medium to operate the Harwood transducer.

Operating Procedure

Removal of Viscometer Cartridge

Open the relief valve on the yellow pump. Open the vent valve at the top of the intensifier about ½ to 1 turn, CCW, with a paper towel under the outlet stem. Unscrew (CCW) the plug nut by hand. Once the threads are disengaged, remove the plug by pulling straight upward. The viscometer cartridge will come out with the vessel plug.

If the vessel will remain open for longer time than is required to replace the sample in the cartridge, the vessel should be filled with pressurizing medium from a syringe or squeeze bottle to prevent air from entering the intensifier region. The compressibility of stray air pockets can limit the pressure capability. If the cartridge will soon be replaced, the contents of the vessel are withdrawn with a syringe using the long blunt needle and replaced with 1 ml of pressurizing medium.

Filling the Viscometer Cartridge

All handling of the cartridge should be done in a pan lined with paper towel. The cartridge is separated from the vessel plug by rotating CCW while holding the white plastic spacer next to the spring at the top of Figure 4.

The cartridge end plug is removed by rotating CCW with a flat screwdriver. With the end plug removed, the previous sample and the presently installed sinker can fall out of the end of the tube onto a paper towel. Any small diameter pushing rod, inserted through the end which previously contained the sinker, is used to push the isolating piston out of the opposite end of the cartridge. Unscrew the fill plug, a 2-56 screw with o-ring seal, from the piston. The fill plug, piston, and end plug each have o-rings and are best cleaned by wiping with a paper towel to prevent solvent damage. The cartridge and sinker are best cleaned in solvent.

Draw about 3 ml of sample into the sample syringe. Drop the selected sinker into the smaller bore end of the cartridge with the top of the sinker going in first as shown in Figure 6. The "H" sinker is marked "T" for top and the "S" and "C" sinkers have an extended point in the center of the bottom surface. Screw the end plug into the bottom of the tube to retain the sinker. Add sample to the open end of the cartridge up to the beginning of the exposed internal threads. Insert the compensating piston, without its plug, until the internal threads of the cartridge are just exposed. The proper orientation of the piston is shown in Figure 7. This is somewhat difficult as the piston o-ring must slide with some interference into the bore. It has been helpful to push the piston in a circular fashion around the edges with a small screwdriver until the o-ring enters the bore.

It is advantageous to remove small air bubbles and dissolved gas before installing the viscometer cartridge. A syringe with a luer-lock fitting with a sealed threaded end and o-ring identical to the fill plug is provided for this purpose and is shown at the top of Figure 8. The glass syringe containing the remaining sample is screwed into the isolating piston where the fill plug will go. With the syringe above the vertical cartridge, the syringe piston is pulled outward until gas bubbles are drawn out of the cartridge, Figure 8. Do this repeatedly until no more bubbles appear. Heating the cartridge in an oil bath or with a heat gun is helpful. Remove the syringe from the top of the cartridge and loosely install the fill plug using the special tool.

The cartridge will be placed vertically on a tabletop with the cartridge continuously pushed against a tabletop surface while the compensating piston is pushed to its starting position and sealed. A special hex wrench is supplied for this purpose, shown at center right in the box of Figure 5. The fill plug is inserted into the compensating piston in the top of the cartridge as shown in Figure 9. The plug is first tightened until the o-ring resistance is encountered and then loosened about ½ to 1 turn CCW to allow some leaking. With the hex wrench still engaged in the fill plug and the end plug pushed against the tabletop push the compensating piston down into the tube using the special hex wrench. More sample must leak from the loose fill plug to accomplish this. Stop when there is about 1 to 2 mm clearance between the top of the tube and the cylindrical handle of the hex wrench, Figure 10. Tighten the fill plug. This position of the isolating piston will allow thermal expansion of the sample on heating and give sufficient clearance during compression of the sample. While holding the tube vertically with the end plug pressed against the tabletop, push down on the fill plug with the special hex wrench. It is very important that you verify by this method that the cartridge is filled with liquid and that no substantial air volume is present by noting that the piston feels "solid" not "spongy". Pressurizing the cartridge when it contains a substantial quantity of air may result in collapse of the tube.

The cartridge is attached to the vessel plug by rotating CW while holding the white plastic spacer next to the spring at the top of Figure 4. If not already done, remove the residual pressurizing liquid from the vessel with a syringe using the long blunt needle and replace with 1 ml of pressurizing medium.

Closing Viscometer Vessel and Charging the System

The valve opening and closing sequences should make reasonably good sense. Think about the consequences of each change in valve configuration before making the change. A venting valve is installed at the top of the intensifier for charging and venting the viscometer. See Figure 11. The trim of the venting valve cannot withstand high pressure (>40 MPa) and this valve must remain closed at high pressure. A small valve has been added to the quick-connect

fitting at the end of the small charging pump hose, shown attached to the venting valve in Figure 11. The purpose of this valve on the charging pump hose is to prevent air from entering the hose when the connection is undone.

Do not put an empty viscometer cartridge into the vessel; pressurizing an empty cartridge will cause it to collapse. Place a piece of paper towel under the exit fitting of the vent valve to absorb the liquid that is released and open the valve ½ to 1 turn CCW to vent air and liquid as the cartridge is inserted. Insert the filled cartridge, attached to the vessel plug, into the vessel. Lightly push down on the nut until the anti-rotation pin rests on the top of the vessel cover. To engage the anti-rotation pin in the internal slot in the vessel cover, push down while rotating the black socket head screw which resides in the vessel plug. Screw the 1 1/16 inch nut CW into the top of the vessel by hand only. Some pressurizing medium will leak from the vent valve onto the towel. The 1 1/16 inch nut should experience a hard-stop when fully tightened. A red dot on the plug nut will face toward the intensifier and a solid stop will be felt when the plug is properly positioned. It is important to check for proper positioning of the plug to ensure that the seals are fully engaged before high (>30 MPa) pressure is applied. Do not tighten the 1¼ inch plug nut. Simply turn it CW until the stop is felt and the red dot faces the intensifier.

Engage the quick-connect fitting at the end of the small hose of the green charging pump with the quick-connect fitting on the vent valve. See Figure 11. Open the shut-off valve at the end of the small hose of the green charging pump. Open the pressure relief valve on the yellow pressurizing pump, close the pressure relief valve on the green charging pump and operate the green pump, at most three strokes, to charge the intensifier with pressurizing liquid (diethylhexyl sebacate). Pressure, as measured by the transducer, of about 5 MPa on the high pressure side is required to move the intensifier piston. The end of the travel of the piston can be felt at the green pump handle. A pressure of about 20 MPa indicates that the intensifier is fully charged. Open the relief valve on the green pump. Close the shut-off valve at the end of the small hose and disengage the quick-connects at the intensifier vent valve. Close the intensifier vent valve (Figure 11) and close the pressure relief valve on the yellow pressurizing pump. The system is now ready for elevated pressure.

Viscometer Operation

The measured temperature should be maintained to within about 1˚C of the testing temperature for at least 15 minutes prior to beginning a measurement. It should be possible to maintain the measured temperature within 0.3˚C of the test temperature.

It is best to begin with the lowest temperature and increase pressure until the fall time becomes inconveniently long or the sample solidifies. Solidification is detected by fall times that increase with time and, for pure liquids, a steady pressure drop of more than the normal relaxation of 2 to 6 MPa following a 50 MPa increase. Solidification of even a small fraction of the sample, as in the case of wax content, will eventually arrest the sinker. Solidification of pure substances can involve a large volume change. For pure liquids, it is necessary to be attentive following a pressure increase and if solidification is detected, reduce the pressure. This instrument is very sensitive to phase separations and with care can be used to generate pressure-temperature phase diagrams and history-dependent pressure-viscosity curves (thixotropy).

If the initial test pressure at a given test temperature is to be ambient pressure, it may be necessary, due to intensifier seal friction and thermal expansion, to open the intensifier vent valve to reduce the indicated pressure to zero (only after reducing the pressure using the yellow pump relief valve!). To bring the pressure to zero, first slowly open the pressure relief valve on the yellow pressurizing pump before opening the vent valve. Reductions in pressure should be done slowly.

A fall of the sinker is reset by rotating the entire viscometer and intensifier about the axis of the intensifier. See Figures 2 and 3. The sinker will travel toward the top of the viscometer as indicated by a rising signal on the oscilloscope display. Once the signal has increased about 500 mV, trigger the scope and rotate the viscometer back to its latched rest position. It is necessary to have the viscometer latched before the signal enters the measurement window – an interval of voltage of from 100 to 300 mV. The trace on the scope display should show a linear descending portion followed by a horizontal portion with only a short transition between the two. See Figure 12. The transition is larger for the hollow sinker. The lower horizontal trace indicates the sinker has stopped at the bottom of the cartridge and is resting on the cartridge end plug. Place a cursor on the linear descending trace about 50 mV above the horizontal trace (150 mV for the hollow sinker) and reset the time and voltage display at that position as shown by the "+" marker in Figure 12. Place the second cursor on the descending trace 100, 200 or 300 mV above the first cursor as seen by the horizontal and vertical lines in Figure 12.

The difference in time between the two cursors is t_f and the difference in voltage divided by 100 mV is the divisor, D. For the example of Figure 12, D=300.00/100=3 and t_f =1.97 s. The fall time is $t = t_f/D$, to be used in equation (1) to calculate the viscosity. The smallest voltage difference for a reasonably accurate measurement is believed to be 100 mV. If convenient, 300 mV or more is definitely preferred. Much larger voltage differences than 500 mV may place the measurement outside of the linear range.

Microsoft Excel programs are provided for each sinker for data reduction and the calculation of pressure-viscosity coefficients. A value for density, ρ, is required in equation (1). The programs that are provided use the temperature modified Tait equation to estimate the temperature and pressure variation of density. It is necessary to provide a value for the density at ambient pressure and the temperature at which that density was measured.

Pressure increments are generally in steps of 100 MPa except after ambient pressure measurement where the pressures are 25 and 50 MPa. For liquids of very high pressure-viscosity coefficient, it may be useful to use 25 MPa pressure steps. Following a pressure increase of 100 MPa, it is natural for the pressure to relax by about 3 to 8 MPa. This relaxation of pressure is due to cooling of the heat of compression. Excessive and continuous loss of pressure is either due to sample solidification which will be apparent in slowing of the fall velocity or a leak. A leak of the intensifier venting valve will be noticed at its quick-connect fitting. A leak of the intensifier piston seals will be noticed at the air vent slot which is at the conjunction of the high pressure cylinder and the low pressure cylinder. The high pressure medium is clear. The oil from the yellow pump which resides on the low pressure side is colored blue. Once a pressure-viscosity isotherm is complete, system pressure should be slowly reduced by slightly opening the vent valve on the yellow pressurizing pump.

Inspection and Replacement of Intensifier Seals

The intensifier received many pressure cycles in this laboratory in the course of calibrations and the measurements to verify operation and the high pressure o-ring was replaced before delivery. A version of this intensifier at Georgia Tech has operated for five years without seal replacement. Should an inspection of the seals be necessary, vent the system to atmospheric pressure and, with the viscometer plug in place and the green pump connected to the intensifier, open the relief valve on the yellow pump and push the intensifier piston to the fully charged

position (system pressure of 20 MPa with the yellow pump relief valve open) using the green charging pump. Remove the 8 socket-head screws from the intensifier and disconnect the large quick-connect fitting which serves as a rotary union from the back of the intensifier. Once the eight screws are loosened, the low pressure cylinder is free to rotate against the high pressure cylinder which facilitates removal of the screws. Prop the pressure transducer with a block that is 1 ½ inch tall so that the low pressure cylinder, when it separates from the intensifier, will clear the upright support of the gray plastic cradle as shown in Figure 13. Remove the low pressure cylinder by pumping the green pump and catch the low pressure cylinder as it is driven out of the high pressure cylinder by the pressurizing liquid. Catch the escaping liquid with paper towels. See Figure 13. Look into the space inside the low pressure cylinder for debris and oil. If there are no debris and the cylinder is relatively dry, begin reassembly.

The small high pressure rubber o-ring will become very rough in service with attached strings of rubber and this is normal. We have not observed wear of the polymer high pressure back-up ring. The high pressure o-ring is removed by removing the retaining washer which is retained by a Phillips-head screw.

Although we have never found this necessary, if the low pressure o-ring is to be replaced, the piston must be removed from the low pressure cylinder. The piston is removed by applying shop air to the back of the low pressure cylinder and catching the ballistic piston in a cardboard box. Replace the large o-ring and push the piston back into the cylinder. This will require release of air from the low pressure cylinder.

Reassemble the intensifier by pouring as much pressurizing liquid as possible into the tilted high pressure cylinder. Push on the low pressure cylinder to drive the seals and piston into the high pressure cylinder with the charging system open to receive the displaced liquid and air. Replace the screws and rock the assembly back into the cradle. Now the system will contain air. Cycle the intensifier piston back and forth with the green and yellow pumps. Be sure that both of the green pump valves are open when operating the yellow pump as operation of the yellow pump can generate sufficient system pressure to fail the charging system. The compressibility of air in the high pressure side will prevent the operation f the viscometer at high pressure.

An Example of the Pressure and the Viscosity Capability

Measurements were performed on a silicone oil, Sigma-Aldrich DMPS-1C lot 89H1168, a dimethyl siloxane of 100cS grade. This is a rather compressible liquid such that the pressure-log viscosity inflection occurs at relatively low pressure (240 MPa). See Figure 14. Two sinkers, the solid and hollow, were necessary to completed the pressure range.

Paraffinic mineral oils will fill precipitate a weak waxy solid at high pressure. The cloud point is pressure dependent. The result is that the viscosity becomes history (and stress) dependent. The history dependence is illustrated in Figure 15 for a Dexron III ATF.

Table 1. Properties of Sinkers

	Sinker "Cup"	Sinker "Solid"	Sinker "Hollow"
Density, ρ_s / g/cm^3	7.75	7.75	7.75
C_0 / mPas/s	3.39	43.6	6100
C_1 / mPas/s/C	-0.00302	-0.037	-7
C_2 / mPas/s/C^2	0	0	0
Shear Stress / Pa	1.3	6	50
Estimated minimum viscosity / mPas	0.05	0.5	300
Calibration Standard	Octane[1]	Cannon S20[2] Diisodecylphthalate sample B[3]	Diisodecylphthalate sample B[4.] Cannon N100[5]
Calibration Factor, $C = C_0 + C_1T + C_2T^2$ T = temperature in ˚C			

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2. KANDIL Mohamed E.; HARRIS Kenneth R.; GOODWIN Anthony R. H.; HSU Kai; MARSH Kenneth N., "Measurement of the Viscosity and Density of a Reference Fluid, with Nominal Viscosity at T = 298 K and p = 0.1 MPa of 29 mPa·s, at Temperatures between (273 and 423) K and Pressures below 275 MPa," J. Chem. Eng. Data, 2006, 51 (6), pp 2185–2196

3. AL MOTARI M.M.; KANDIL Mohamed E.; MARSH Kenneth N. GOODWIN Anthony R. H., "Density and Viscosity of Diisodecyl Phthalte…," J. Chem. Eng. Data, 2007, 52, pp 1233–1239

4. Viscosities measured at elevated pressure at 30, 50, 75 and 100C using the "S" sinker.

5. Cannon data

Table 2. Temperature control settings in Atlanta

Test Temperature/C	Heater Voltage	Target Air Temperature/C
30	29	31.3
40	39	42.9
50	52	54.7
75	73	84.5
80	75	89.8
100	82	114.1
125	92	143.1
150	103	175

Table 3. Intensifier calibrations

Low Side	Heise	Reading	Difference
psi	MPa	MPa	MPa
0	0	0	
1020	100	100	0
2000	200	201	1
2980	300	302	2
3960	400*	403	3

* When directly compared, a Harwood Manganin Cell (Model C7186) indicates 3 MPa greater pressure than does the Heise gauge at 400MPa.

Table 4. O-ring Specifications

Position	Type	Source
Vessel plug	-010-70 BN	Hardware Store
Cartridge End Plug	-007-70 BN	Hardware Store
Cartridge Compensating Piston	-008-70 BN	Hardware Store
Intensifier Piston-Small End	.354 x .087 – 70 BN	Apple Rubber Prod.
Intensifier Piston-Large End	-224-70 BN	Hydraulic Specialty
Fill Plug	.079 x .024 – 70 BN	Apple Rubber Prod.
Syringe Fitting	.079 x .024 – 70 BN	Apple Rubber Prod.

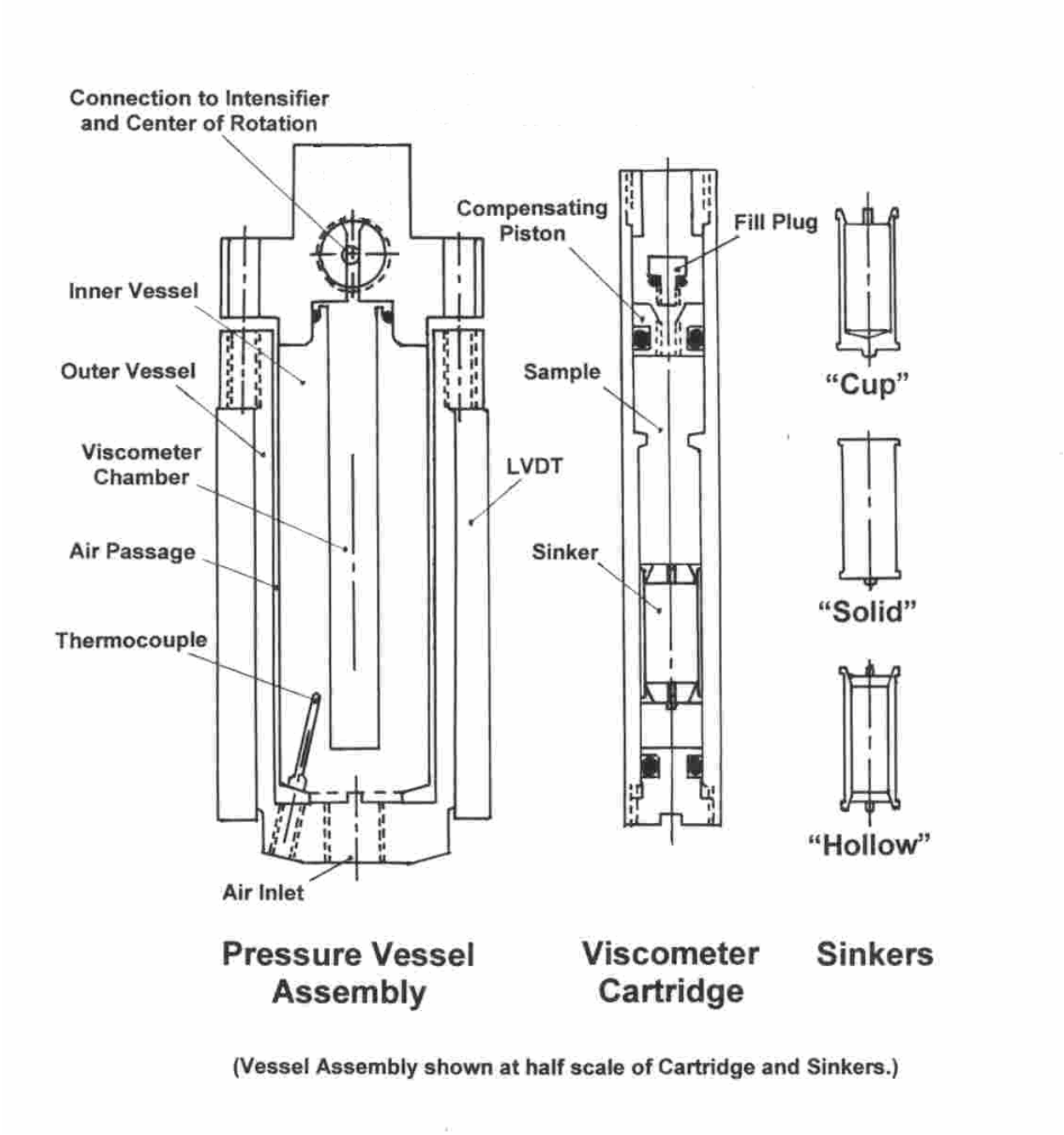

Figure 1. Viscometer cross-section. Modifications to the upper part of the vessel have not been reproduced in this figure.

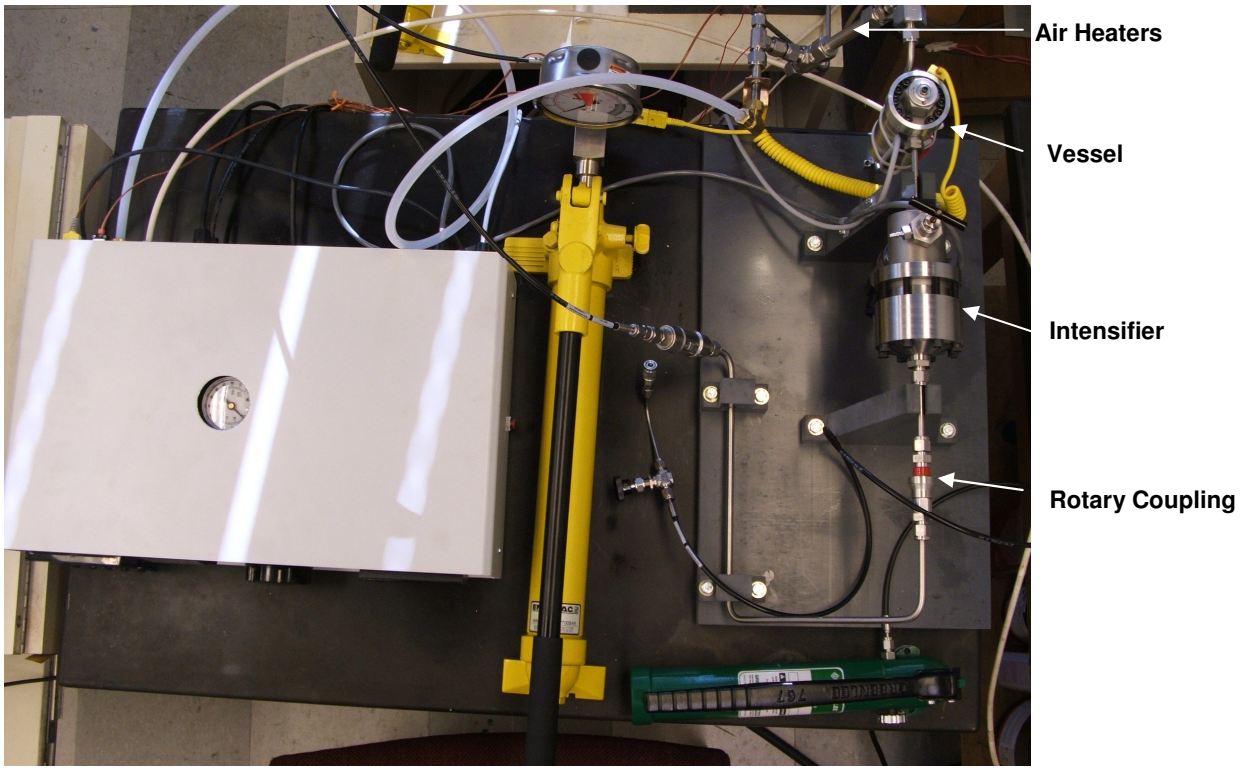

Figure 2. Viscometer and associated components, oscilloscope not shown.

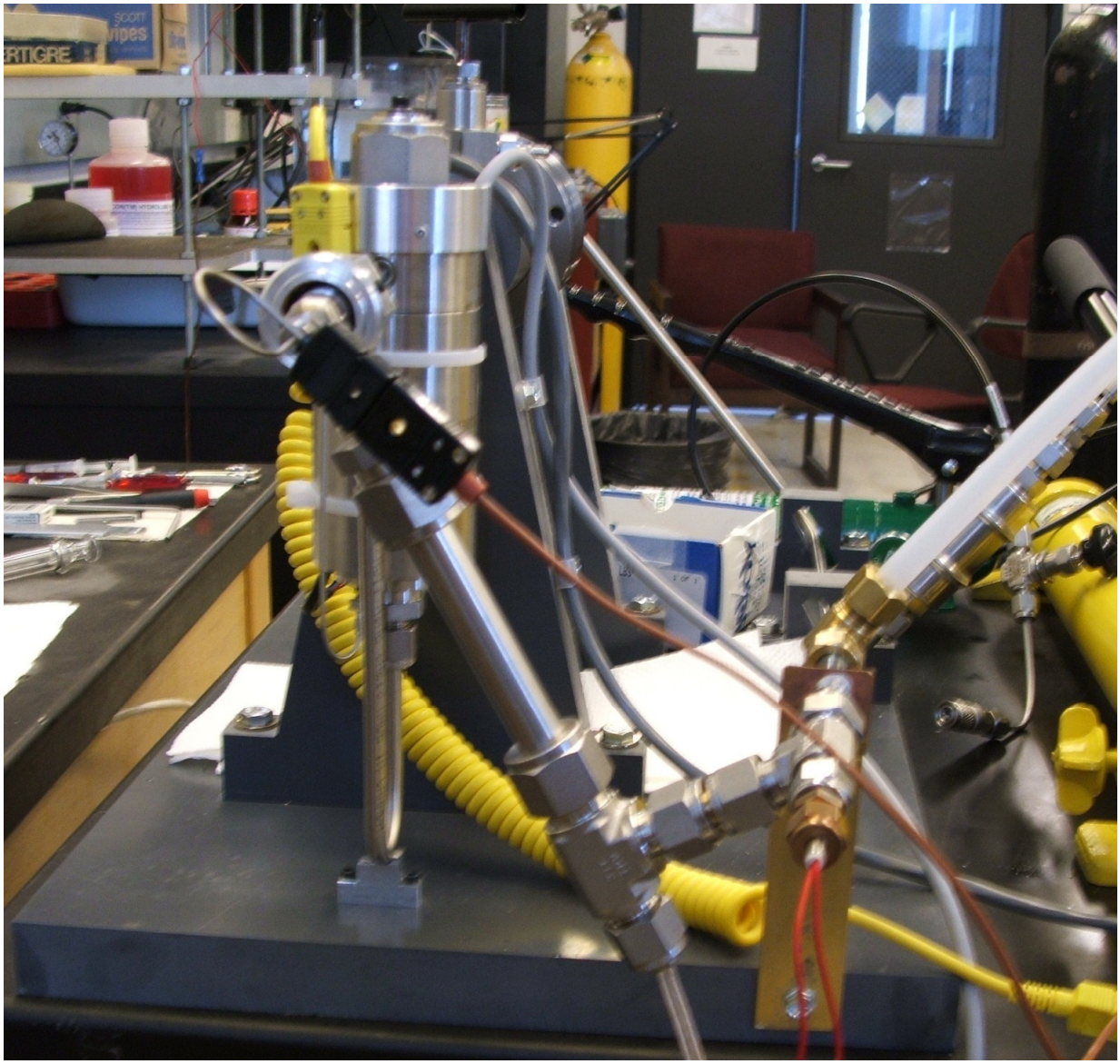

Figure 3. The air heating system.

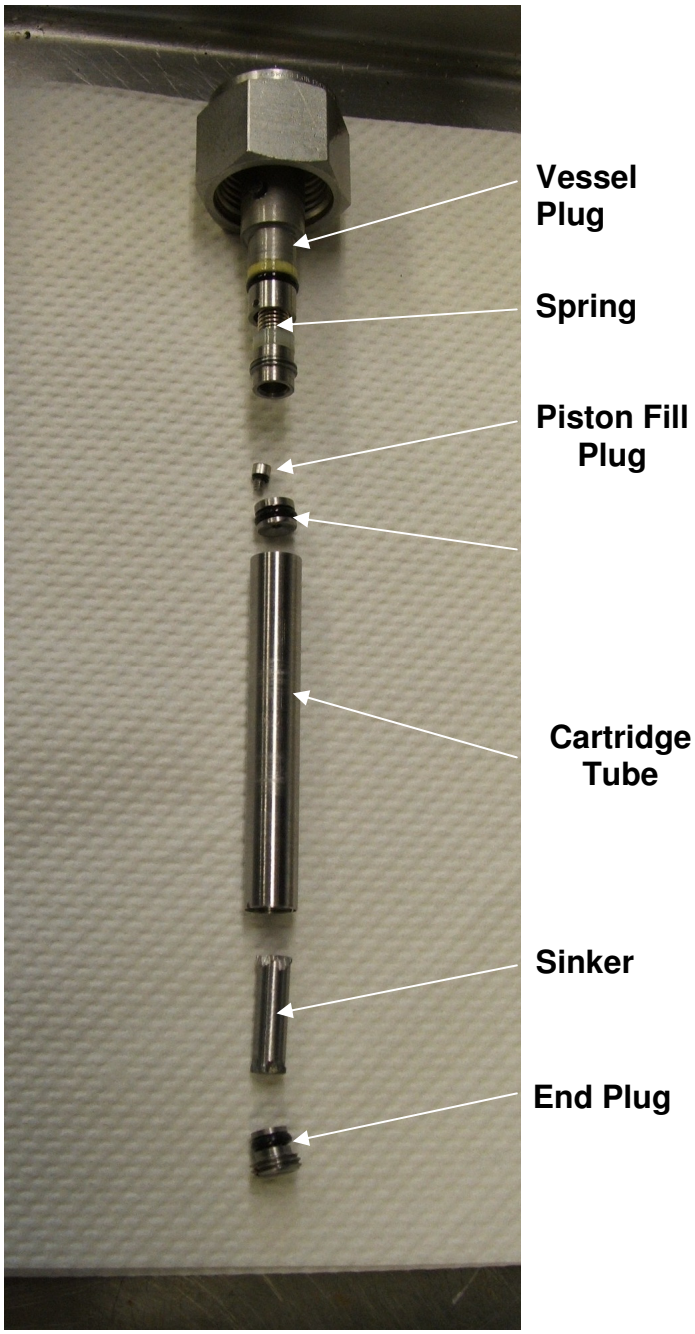

Figure 4. Viscometer cartridge components. From top to bottom: vessel plug with seal and spring, compensating piston plug, compensating piston, cartridge tube, sinker, and cartridge plug.

Figure 5. Box of cartridge parts and tools.

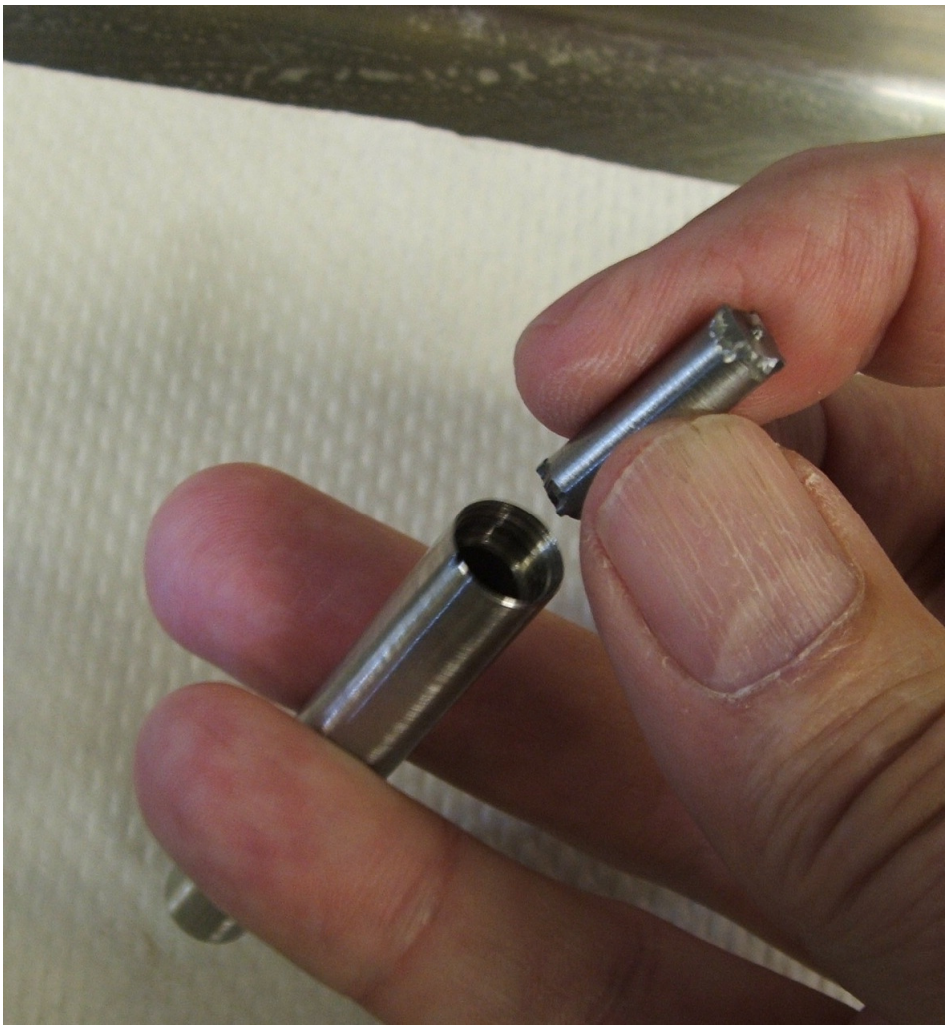

Figure 6. Insert the sinker with "T" entering first or bumper point (as shown) entering last.

Figure 7. Insert the compensating piston without the fill plug and push in to just past the threads at the end of the cartridge.

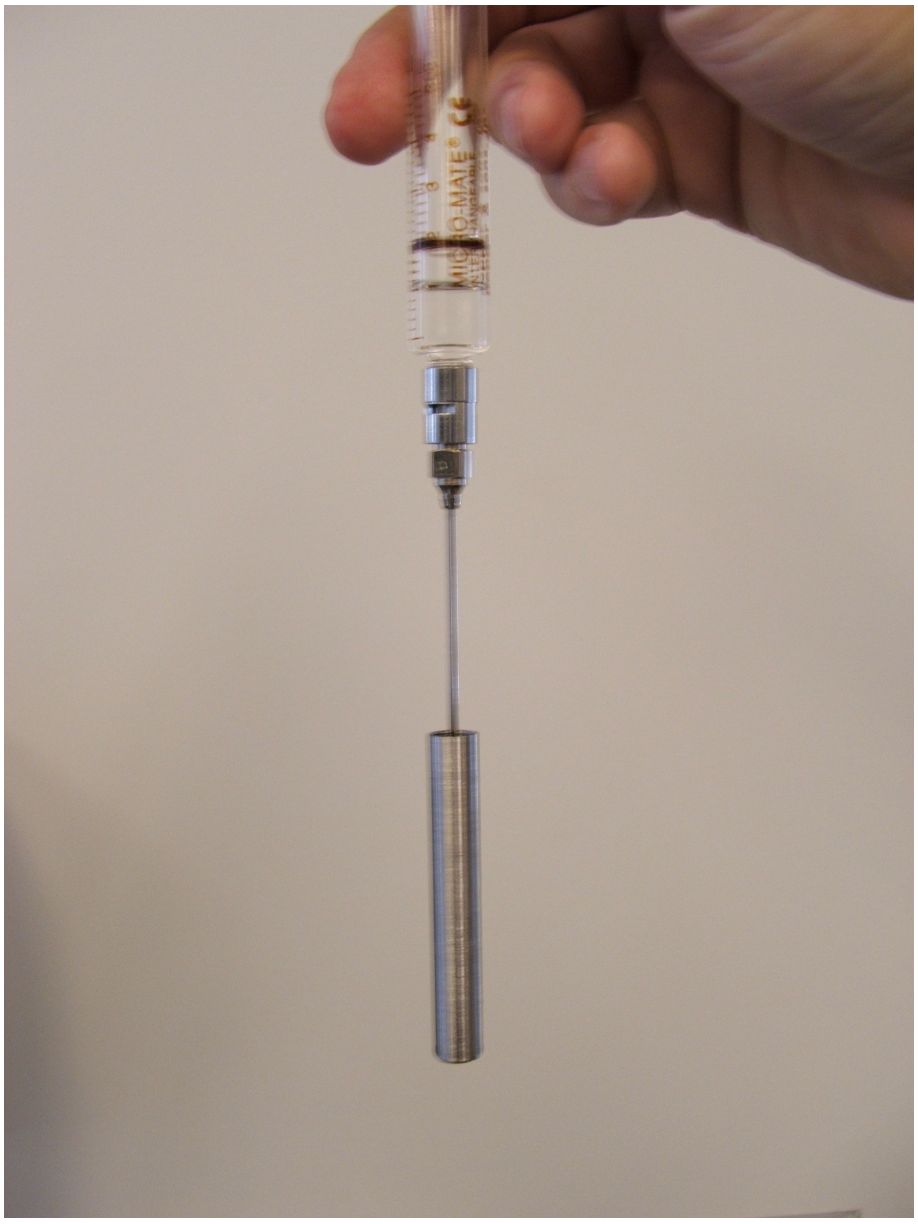

Figure 8. Withdraw bubbles with syringe. Heating by air gun or oil bath is helpful.

Figure 9. Insert the end plug.

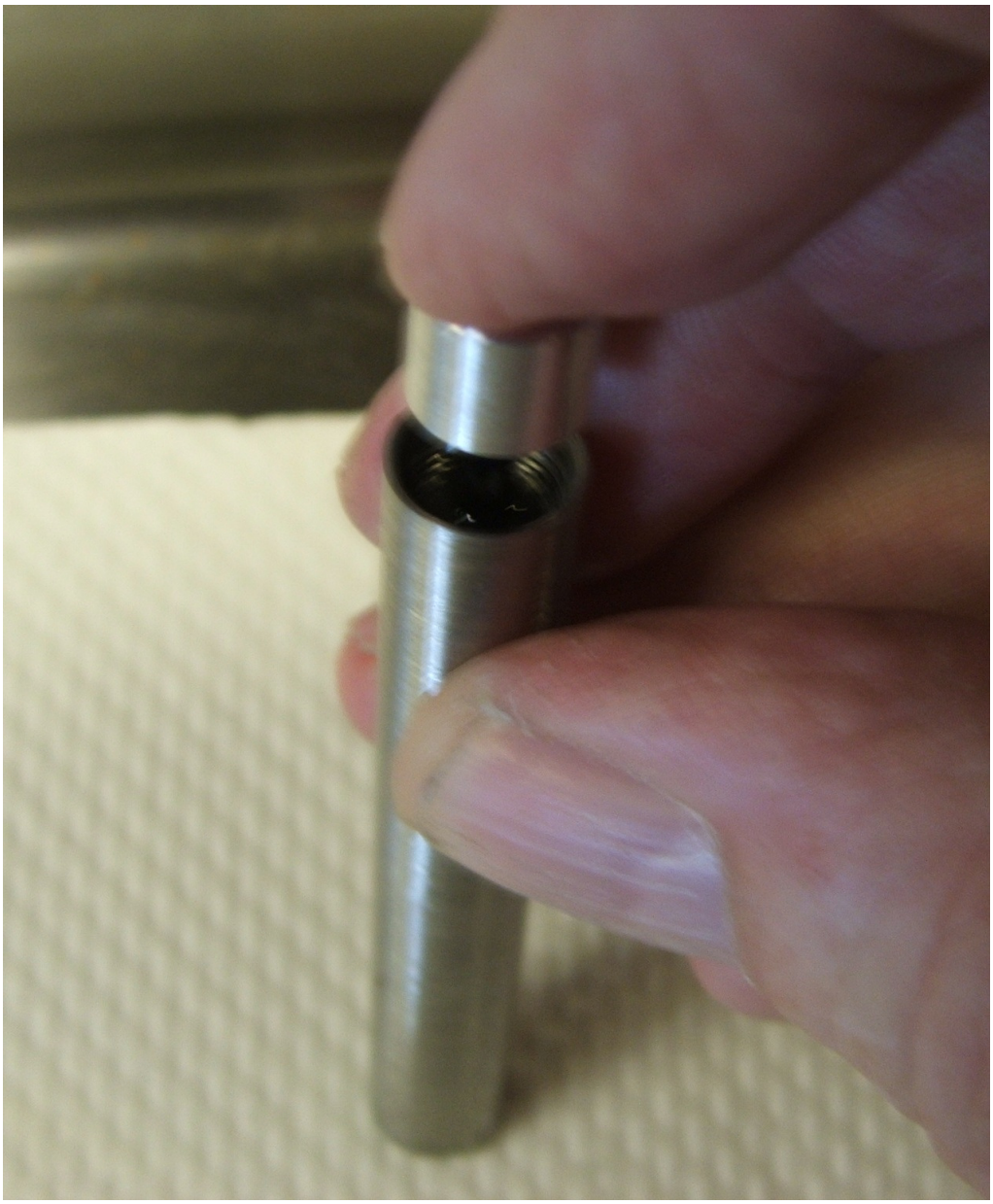

Figure 10. Push the compensating piston into the cartridge with the special hex wrench.

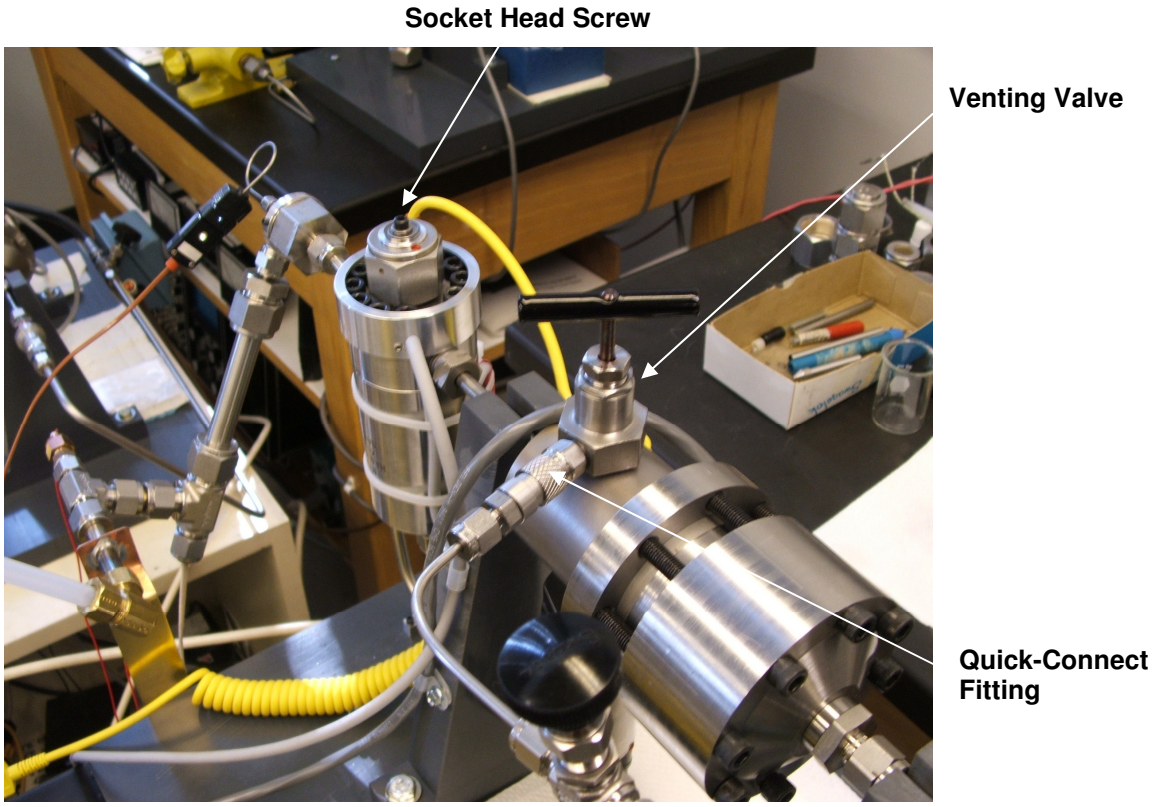

Figure 11. A venting and charging valve resides at the top of the intensifier. It may be connected to the green charging pump by a quick-connect fitting.

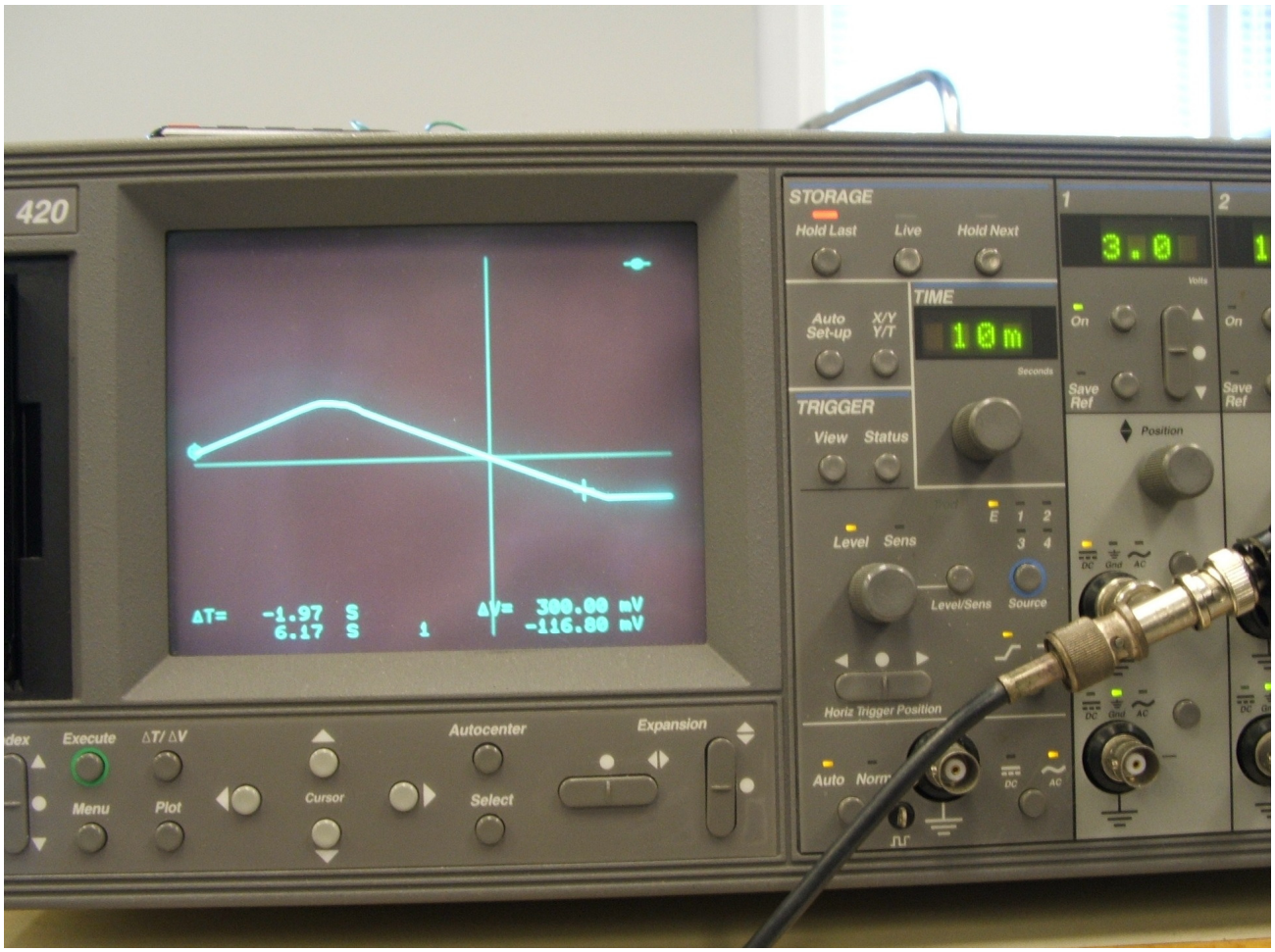

Figure 12. The oscilloscope trace for a successful fall of the sinker.

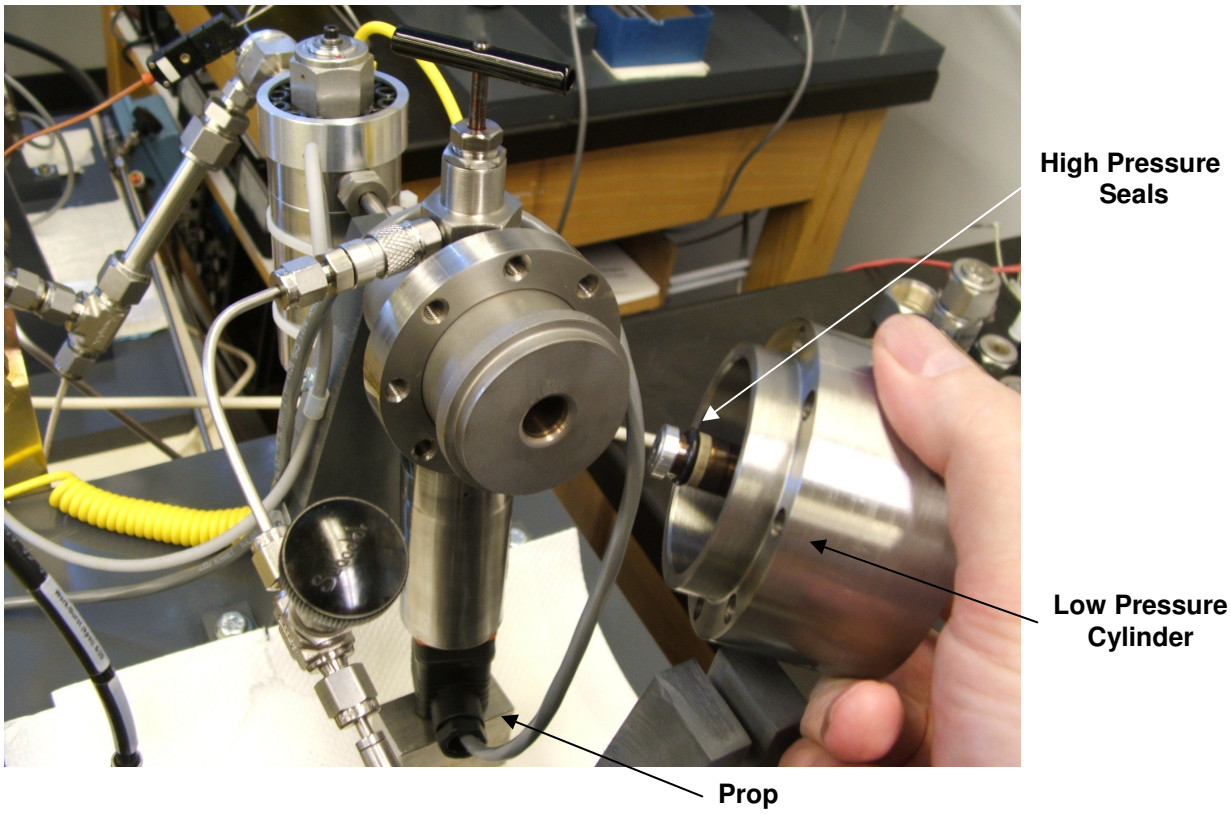

Figure 13. Removing the piston and seals from the high pressure cylinder. The low pressure cylinder containing the intensifier piston is at lower right. The small hose from the green pump is shown attached to the intensifier venting valve at the top.

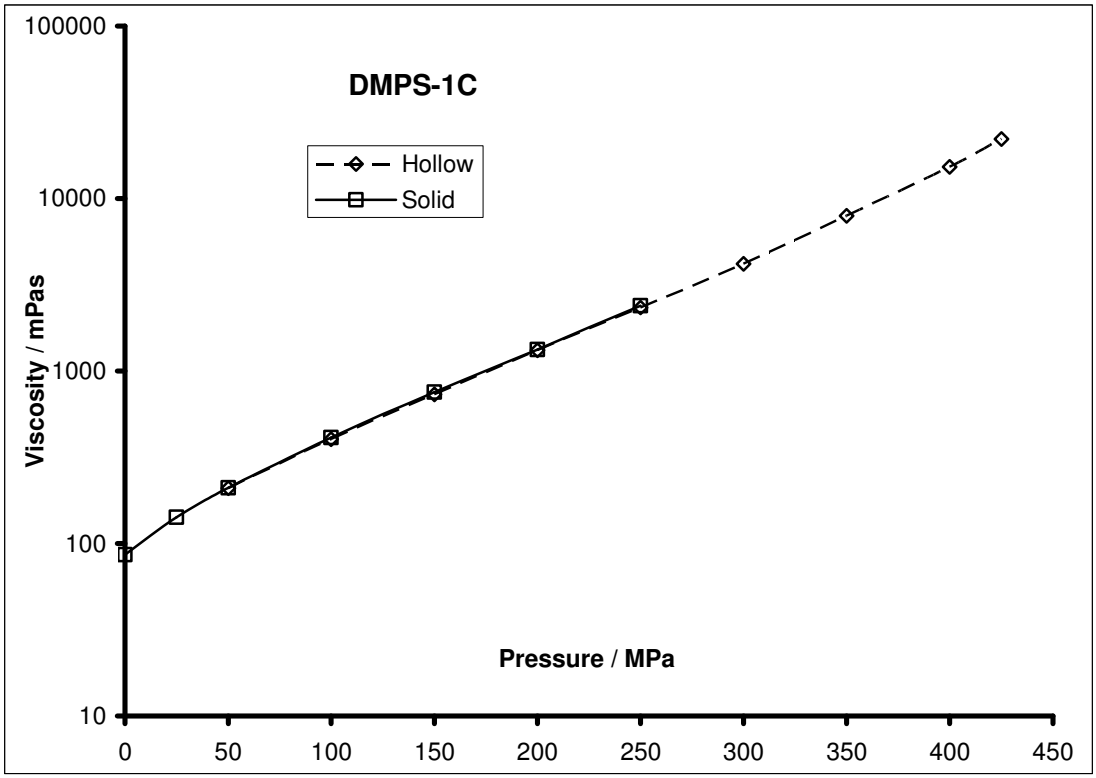

Figure 14. An example of the pressure and viscosity capability.

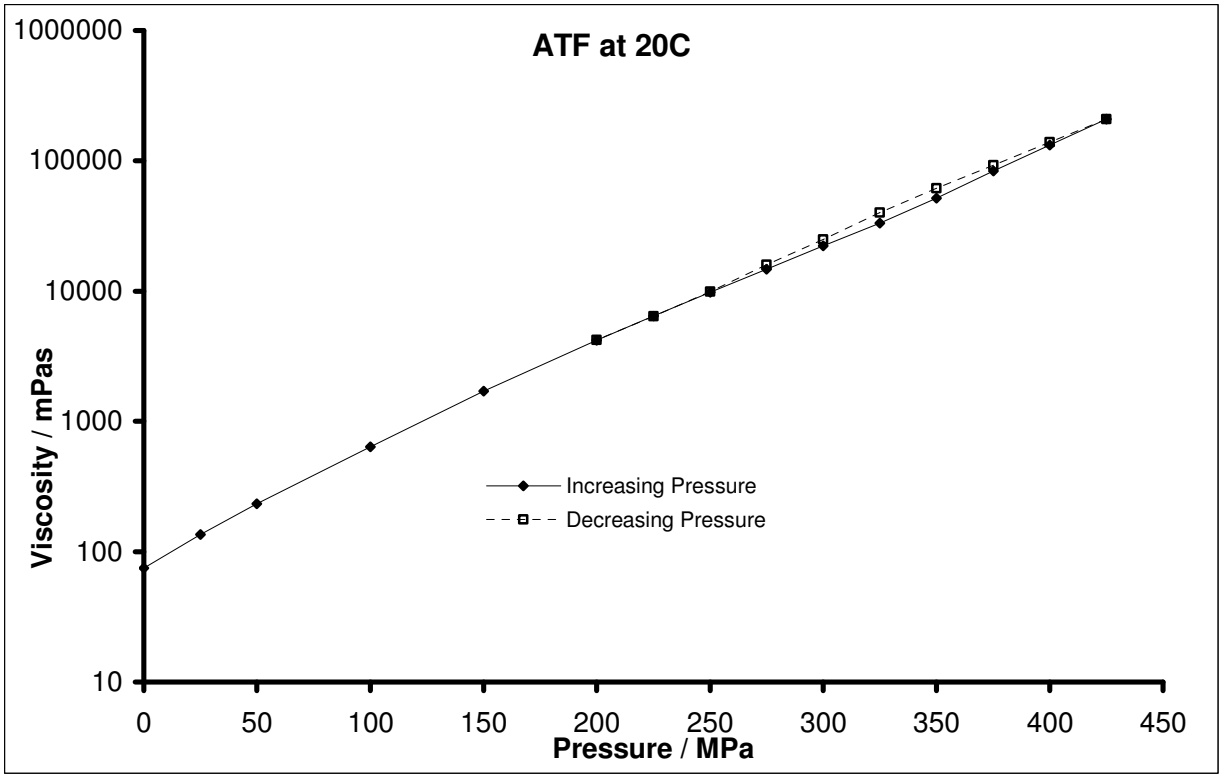

Figure 15. An example of thixotropy in a mineral oil based ATF.